



Codex Methods of Analysis: What, When, Why, How to Use?

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Codex methods of analysis and quality assurance provisions – brief survey of the requirements introduced and their rationale

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Need to Cover Today:

General Principle on Codex Methods of Analysis

- Fully validated (GL 64)
- Single laboratory validated (GL 49)

Significance of Typing of Methods - Methods in Legislation

- Performance Parameters
- Requirements for the *Codex Alimentarius* Method Performance Criteria approach

Laboratory Quality Standards

- GL 27
- IUPAC Proficiency Testing Protocol
- IUPAC Internal Quality Control (GL 65)
- IUPAC Recovery Guidelines (GL 37)



Method Validation

Full validation of methods – collaborative trial requirements

Verification of the performance of standardised methods in an individual laboratory

Single Laboratory Validation

Assessment of Precision Characteristics

Internal Quality Control (IQC) in Laboratory Quality Assurance Programmes

Introduction to IQC

IQC Procedure & Quality Assurance Practices

Recommendations



Role of Proficiency Testing in Assessment of Laboratory Quality

Measurement Uncertainty

Introduction to Estimation of Measurement Uncertainty

Various Approaches : ISO/EURACHEM, CODEX

Conclusions

Recovery Corrections - Do We Need to Carry Out?

Introduction to Recovery Corrections

Uncertainty in Reporting Recovery

Recommendations



Guidelines for settling disputes over analytical (test) results
(CAC/GL 70-2009)

Guidelines on analytical terminology
(CAC/GL 72-2009)



Codex relationship provisions: “The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards”

How we interpret the results – measurement uncertainty

MU guidelines (GL 54)

Compliance and measurement uncertainty



CODEX ALIMENTARIUS COMMISSION

Principles for the establishment of Codex methods of analysis

The methods are primarily intended as international methods for the verification of provisions in Codex standards. They should be used for reference, in calibration of methods in use or introduced for routine examination and control purposes.



Definition of types of methods of analysis

Defining Methods (Type I)

Definition: A method which determines a value that can only be arrived at in terms of the method per se and serves by definition as the only method for establishing the accepted value of the item measured.

Examples: Howard Mould Count, Reichert-Meissl value, loss on drying, salt in brine by density.



Reference Methods (Type II)

Definition: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). It should be recommended for use in cases of dispute and for calibration purposes.

Example: Potentiometric method for halides.



Alternative Approved Methods (Type III)

Definition: A Type III Method is one which meets the criteria required by the Codex Committee on Methods of Analysis and Sampling for methods that may be used for control, inspection or regulatory purposes.

Example: Volhard Method or Mohr Method for chlorides



Tentative Method (Type IV)

Definition: A Type IV Method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Codex Committee on Methods of Analysis and Sampling have not yet been determined.

Examples: chlorine by X-ray fluorescence, estimation of synthetic colours in foods.



General Criteria for the Selection of Methods of Analysis

Official methods of analysis elaborated by international organizations occupying themselves with a food or group of foods should be preferred.

Preference should be given to methods of analysis the reliability of which have been established in respect of the following criteria, selected as appropriate:

- Specificity
- Accuracy



- Precision – repeatability intra-laboratory (within laboratory),
- Precision - reproducibility inter-laboratory (within laboratory and between laboratories)
- Limit of detection
- Sensitivity
- Practicability and applicability under normal laboratory conditions
- Other criteria which may be selected as required.



WHAT IS THE CRITERIA APPROACH TO METHODS OF ANALYSIS?

WHY INTRODUCE IT?

What these criteria mean



Traditional Approach (prescribing a specific method of analysis) means:

- The analyst is denied freedom of choice and thus may be required to use an inappropriate method in some situations;
- The procedure inhibits the use of automation; and
- It is administratively difficult to change a method found to be unsatisfactory or inferior to another currently available.



Traditional Approach (prescribing a specific method of analysis) does:

apply to Codex Type I, II and III methods

and where the method should be “fully validated”.



Criteria Approach (prescribing performance characteristics) means:

- giving greater flexibility than the present procedure adopted by organisations such as Codex and the EU
- not being in the situation of having many methods of analysis which are available, which meet requirements as regards method performance characteristics, but which are not considered by Codex or the EU because of time constraints.



Only applicable to rationale methods, not to empirical methods (i.e. where the result is method dependent)

Need to define these better?

METHOD CRITERIA



Conversion of Specific Methods of Analysis to Method Criteria by the CCMAS

When a Codex Commodity Committee submits a Type II or Type III method to CCMAS for endorsement, it should also submit information on the specified Codex level(s) along with the provision to enable the CCMAS to convert it into suitable generalized analytical characteristics:

- Trueness
- Applicability (matrix, concentration range and preference given to 'general' methods)

- Limit of detection
- Limit of quantification
- Precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories), but generated from collaborative trial data rather than measurement uncertainty considerations
- Recovery
- Selectivity
- Sensitivity
- Linearity





How Should Operate (1)

A commodity committee should, whenever possible, provide information to the Committee on Methods of Analysis and Sampling for each individual analytical method proposed, relating to specificity, accuracy, precision (repeatability, reproducibility) limit of detection, sensitivity, applicability and practicability, as appropriate. Similarly a commodity committee should, whenever possible, provide information to the Committee on Methods of Analysis and Sampling for each sampling plan relating to the scope or field of application, the type of sampling (e.g. bulk or unit), sample sizes, decision rules, details of plans (e.g. "Operating characteristic" curves), inferences to be made to lots or processes, levels of risk to be accepted and pertinent supportive data.



How Should Operate (2)

The Committee on Methods of Analysis and Sampling will assess the actual analytical performance of the method which has been determined in its validation. This will take account of the appropriate precision characteristics obtained in collaborative trials which may have been carried out on the method together with results from other development work carried out during the course of the method development. The set of criteria that are developed will form part of the report of the endorsement by the Committee on Methods of Analysis and Sampling and will be inserted in the appropriate Codex commodity standard.



How Should Operate (3)

In addition, the Committee on Methods of Analysis and Sampling will identify numeric values for the criteria for which it would wish such methods to comply.

Difficult to do retrospectively – need real examples to go into STAN 234.



GUIDELINES FOR THE ASSESSMENT OF THE COMPETENCE OF TESTING LABORATORIES INVOLVED IN THE IMPORT AND EXPORT CONTROL OF FOOD

Adopted by the Commission at Step 8 in June 1997



SCOPE

1. These guidelines provide a framework for the implementation of quality assurance measures to ensure the competence of testing laboratories involved in the import and export control of foods.
2. These guidelines are intended to assist countries in the application of requirement for trade in foodstuffs in order to protect the consumers and to facilitate fair trade.



REQUIREMENTS

3. The following criteria shall be adopted by laboratories involved in the import and export control of foods:
 - Compliance with the general criteria for testing laboratories laid down in ISO/IEC Guide 25: 1990 "General requirements for the competence of calibration and testing laboratories"; [i.e. effectively accreditation],
 - Participation in appropriate proficiency testing schemes for food analysis which conform to the requirements laid down in "The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories", Pure and Applied Chemistry 65 (1993) 2132-2144; [already adopted for Codex purposes by the CAC at its 21st Session in July 1995]



- Whenever available, use methods of analysis which have been validated according to the principles laid down by the CAC, and
 - Use internal quality control procedures, such as those described in the “Harmonised Guidelines for Internal Quality Control in Analytical Chemistry Laboratories”, Pure and Applied Chemistry 67 (1995) 649-666
4. The bodies assessing the laboratories referred to above should comply with the general criteria for laboratory accreditation, such as those laid down in the ISO/IEC Guide 58:1993: “Calibration and testing laboratory accreditation systems - General requirements for operation and recognition”.



What do these mean?

- Accreditation
- Proficiency Testing
- Internal quality control
- Method validation

Others not mentioned above:

- Recovery
- Single laboratory validation



Also identified in the Codex Alimentarius Commission Procedural Manual

Here a section on:

“The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards”

has been approved and is included in Procedural Manual



ISSUES INVOLVED

There are a number of analytical and sampling considerations which prevent the uniform implementation of legislative standards. In particular, different approaches may be taken regarding sampling procedures, the use of measurement uncertainty and recovery corrections. At present there is no official guidance on how to interpret analytical results across the Codex Community. Significantly different decisions may be taken after analysis of the “same sample”. For example some countries use an “every-item-must-comply” sampling regime, others use an “average of a lot” regime, some deduct the measurement uncertainty associated with the result, others do not, some countries correct analytical results for recovery, others do not. This interpretation may also be affected by the number of significant figures included in any commodity specification.



It is essential analytical results are interpreted in the same way if there is to be equivalence across the Codex Community.

It is stressed that this is not an analysis or sampling problem as such but an administrative problem which has been highlighted as the result of recent activities in the analytical sector, most notably the development of International Guidelines on the Use of Recovery Factors when Reporting Analytical Results and various Guides prepared dealing with Measurement Uncertainty.



RECOMMENDATIONS

It is recommended that when a Codex Commodity Committee discusses and agrees on a commodity specification and the analytical methods concerned, it states the following information in the Codex Standard:



1. Sampling Plans

The appropriate sampling plan to control conformity of products with the specification. This should state:

- whether the specification applies to every item in a lot, to the average in a lot or the proportion nonconforming;
- the appropriate acceptable quality level to be used;
- the acceptance conditions of a lot controlled, in relation to the qualitative/quantitative characteristic determined on the sample.



2. Measurement Uncertainty

That an allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification. This requirement may not apply in situations when a direct health hazard is concerned, such as for food pathogens.



3. Recovery

Where relevant and appropriate the analytical results are to be reported on a recovery corrected basis and that the recovery should be quoted in any analytical report. Analytical results are to be expressed on a recovery corrected basis where appropriate and relevant, **and when corrected it has to be so stated.**

In all cases it has to be stated when the result is corrected for recovery.



If a result has been corrected for recovery, the method by which the recovery was taken into account should be stated. The recovery rate is to be quoted **wherever** possible.

When laying down provisions for standards, it will be necessary to state whether the result obtained by a method used for analysis within conformity checks shall be expressed on an recovery-corrected basis or not.



4. Significant Figures

The units in which the results are to be expressed and the number of significant figures to be included in the reported result.